25p

MTP-ASTR-P-63-16
November 19, 1963

OTS: $

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HUNTSVILLE, ALABAMA

(NASA TMX 54615)

EFFECTS OF GAMMA RADIATION ON SELECTED POTTING COMPOUNDS AND INSULATING MATERIALS

by

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ABSTRACT

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It was found that at the maximum doses to which the subject material was exposed, Mylar and polyester films performed well. Teflon performed very poorly under the same conditions. Among the potting compounds, the epoxy resins showed the effects induced by both chain scission and crosslinking. The silicones changed only slightly in physical properties. Test data indicated a gradual degradation in the properties of polyurethane resins.

This report represents the initial portion of a continuing effort in the evaluation of polymeric materials for use in radiation environments. Present plans call for more extensive testing in combined space environments in the near future.

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EFFECTS OF GAMMA RADIATION ON SELECTED POTTING COMPOUNDS AND INSULATING MATERIALS

by

Bobby W. Kennedy

Electrical Engineering Development Section
Pilot Manufacturing and Development Branch
Astrionics Laboratory

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SUMMARY

Recent radiation tests were performed at the Oak Ridge National Laboratory on the following types of polymeric materials: epoxy, silicone, and polyurethane resins; teflon, polyester film, and Mylar flat-conductor cables. After exposures of up to 10^8 roentgens, mechanical properties were analyzed by the Pilot Manufacturing Branch of Astrionics Laboratory. Property changes data were then correlated with molecular effects caused by absorption of gamma rays. How radiation affects the physical properties of common polymeric materials and the specific material irradiated at Oak Ridge are discussed.

It was found that at the maximum doses to which the subject material was exposed, Mylar and polyester films performed well. Teflon performed very poorly under the same conditions. Among the potting compounds, the epoxy resins showed the effects induced by both chain scission and crosslinking. The silicones changed only slightly in physical properties. Test data indicated a gradual degradation in the properties of polyurethane resins.

This report represents the initial portion of a continuing effort in the evaluation of polymeric materials for use in radiation environments. Present plans call for more extensive testing in combined space environments in the near future.

SECTION I. INTRODUCTION

The Pilot Manufacturing and Development Branch of the Astrionics Laboratory is responsible for the selection of polymeric materials for future guidance and control systems. The effects of radiation on materials have taken on importance in recent years because of the development of nuclear-powered rockets and the possibility of extended applications in space environments. The materials discussed include organics used for potting electrical equipment and for insulating conductors. The series of tests discussed represent the initial stage of a larger program of radiation testing.

Gratitude is expressed to Roy E. Currie and W. T. White of the RIFT/ NOVA Office, Astrionics Laboratory, for making arrangements for the current series of radiation tests and for coordinating future activities in radiation testing. Also acknowledgement is made to John R. Rogers of the Design Guidelines Unit, Brown Engineering Co., Inc., who assisted with the writing and interpreting of the data reported.

The radiation effect on organic materials and particularly on how radiation affects physical properties is discussed. An evaluation of the use of physical properties in making predictions concerning molecular changes is made. A survey of the literature indicates that certain plastics tend toward crosslinking while others tend to degrade by scission. Suggestions are made for methods of developing radiation resistant systems.

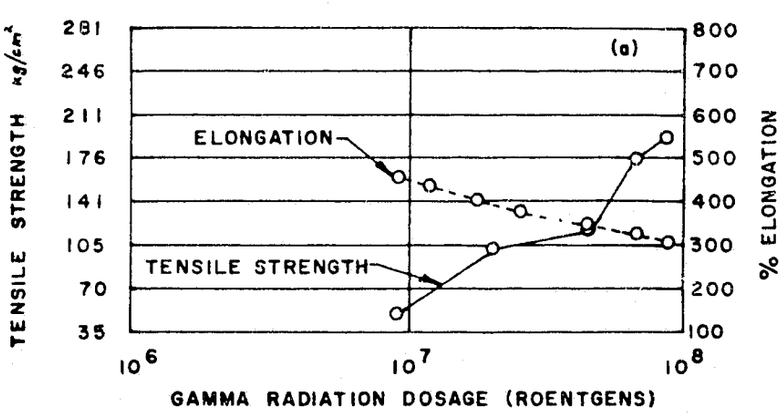
Tables and graphs show the effects of radiation on the particular materials that were recently tested at Oak Ridge. A brief discussion of trends and their possible meanings is included for each class of materials.

SECTION II. CORRELATION OF RADIATION EFFECTS WITH PHYSICAL PROPERTIES

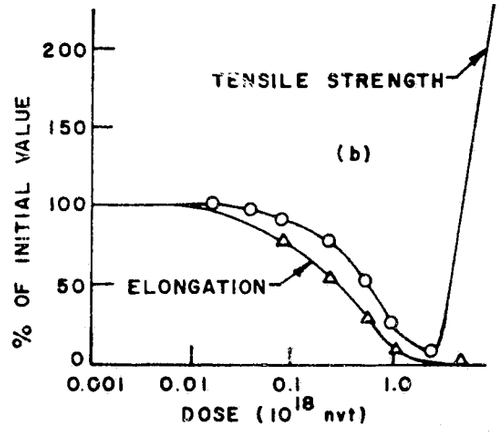
An attempt to evaluate the radiation damage mechanism on the basis of physical property changes is desirable, but must be undertaken with caution. The mechanical property changes of compression strength, hardness, tensile strength, elongation, and breaking strength depend on a number of factors, including molecular weight, degree of branching, amount of crystallinity, extent of crosslinking, and orientation of molecules. It is desirable to stabilize, through consideration of the chemistry of the particular polymer, as many variables as possible. Only then can property changes be correlated with molecular changes to any degree of accuracy. It is clear that predictions regarding the

nature of molecular changes can not be based solely on physical property changes. For example, crosslinking draws molecules closer together and increases hardness, so that the resultant material is brittle and glassy. Scission usually causes opposite changes in physical properties by breaking the molecule into smaller fragments, decreasing the molecular weight, and lowering the melting point. The resultant product becomes soft. However, sometimes crystallinity can be increased by polymers that undergo scission because there is less restraint on the shortened molecule. Thus the molecules become more easily oriented in the crystal structure. An increase in crystallinity is analogous to an increase in molecular weight and the resultant product is similar to that of a polymer that undergoes crosslinking.

In natural rubber when crosslinking predominates, tensile strengths sometime increase to a maximum (Fig. 1a) as the polymer reaches the brittle or resinous stage. The initial maximum may not occur, however, and a steady increase in crosslinking density may be accompanied by a steady decrease in tensile strength and breaking elongation (Fig. 1b).



1a - Gamma Radiation (R) on Natural Rubber (uncured)



1b - Neutron Radiation of Natural Rubber (cured with sulfur)

FIGURE 1. RADIATION EFFECTS ON NATURAL RUBBER

In both cases, the molecular effect is caused by crosslinking, even though the gross effects on tensile strength are diametrically opposed. Thus an interpretation of molecular phenomena based on evidence from tensile strength alone does not prove the dominance of crosslinking over scission or vice versa.

In spite of the difficulties outlined, it is useful to generalize concerning the effects of radiation on materials. For instance, it has been found empirically that polymers react according to the scheme shown in Table I. These materials react, as indicated by changes in the mechanical properties, as follows:

(1) Crosslinking changes rubbery or plastic materials into hard, ultimately brittle solids.

(2) Scission of the molecular chains results in a decrease in average molecular weight and tensile strength and in an increase in elongation.

Table I. Effect of High-Energy Electrons on Polymers (in Air)

Polymers become crosslinked	Polymers degrade by scission
Polystyrene	Polymethyl methacrylate (Plexiglas)
Polyesters	Polyvinylchloride
Polyethylene terephthalate (Mylar)	Polytetrafluoroethylene (Teflon)
Polyethylene	
Hexamethylene diamine (Nylon)	
Natural rubber	

The classification shown in Table I is not entirely rigid, since some polymers may fall on one side or the other depending on the conditions of testing. For example, the presence of oxygen tends to encourage scission; and certain polymers may show predominant scission or crosslinking, depending on whether oxygen is present. Research work has shown that ordinarily vacuum will offer some degree of protection to polymeric materials against damage caused by ionizing radiation. Similarly it is apparent that thick samples or samples immersed in liquid will not react readily because of the absence of oxygen from the site of reaction. The effects of temperature, dose rate of radiation, and hard vacuum may change the rate and direction of reactions in a radiation environment. These variables will be considered in the future testing program.

Developing radiation resistant systems. The organic systems used for encapsulating and potting are usually highly crosslinked systems. Damage is caused by the chemical breakdown of this network. Resins containing radiation resistant groups such as the phenyl group will resist chemical changes best. Phenolics, epoxies, and silicones containing high phenyl/methyl ratios, and resins cured with styren, have superior radiation resistance. In silicones, the Si-O network is highly radiation resistant. In epoxies and polyesters, the network bonds are slowly broken by radiation at a rate dependent on the exact chemical composition.

All organics will evolve some gas because of radiation, but as long as the gases are limited to CO_2, H_2, O_2, and N_2, they probably will not be injurious to men and equipment. In the case of halogenated plastics, commonly used for fire-retardant systems, the designer should beware of the outgassing of strong acid vapors or free halogens.

SECTION III. CURRENT TEST PROGRAM

The radiation tests discussed were carried out at the Atomic Energy Commission's facilities at Oak Ridge, Tennessee. The source of radiation was Cobalt 60 of 17,000 curies of gamma radiation. The dose rate was 1×10^6R per hour at an ambient temperature of 25° C in an atmosphere of air.

The test procedure used with the subject samples consisted of irradiating samples at doses ranging up to 10^8R. Since the dose rate was 10^6R per hour, it is obvious that 10^7R required 10 hours and 10^8R required 100 hours; no test lasted over 100 hours. Following irradiation, various mechanical tests were performed on the materials by the Pilot Manufacturing Branch; comparisons were made between the materials radiated at different levels. Trends could then be correlated on tables and graphs and evaluated.

It is anticipated that future studies will include radiation tests at 25° C and 760 mm Hg and also at high vacuum and cryogenic temperatures. Following the testing in environmental extremes of temperature, vacuum, and radiation, studies will be made on the mechanical properties and also on electrical properties, including dielectric strength, volume resistivity, dissipation factor, and insulation resistance.

The materials selected for study included polymers of the following categories: (1) polyester films, (2) Mylar cables, (3) Teflon film and tubing, (4) epoxy resins, (5) silicone resins, and (6) polyurethane resins.

The following general observations were made after exposing the materials to radiation (Figs. 2 and 3):

(1) At 10^6R, the Permacel pressure-sensitive adhesive used to bond copper foil to metal surfaces was still tacky and exhibited its original bond strength; at 10^8R, the Permacel adhesives had lost all of their peel strength.

(2) At 10^8R, the polyethylene bag that contained samples was shattered by radiation.

(3) Teflon insulation on wire had peeled off at 10^8R; whereas, at 10^6R the Teflon film was too brittle to handle. Teflon samples exhibited no weight loss because of irradiation at 10^5R and 10^6R.

A. POLYMERIC MATERIALS

Three types of polyester films were irradiated during the current series of tests. In addition, two types of flat-conductor cables made of Mylar (polyethylene terephthalate) were irradiated. Test parameters included elongation, breaking load, and tensile strength. According to information presented previously, the irradiation of polyester should result in crosslinking. Therefore, an increase in molecular weight, decrease in tensile strength and in elongation, and an increase in hardness in most of the test samples should be expected (Fig. 1b).

1. Polyester films. There were three different types of polyester films exposed to radiation environments. These were designated A, C, and D. Tables II, III, and IV show the results of testing these materials in different quantities of radiation.

Table II. Radiation Effects on Polyester Film

Spl. No.	Total Dose (R)	No. of Specs.	Width cm (in.)	Thickness cm (in.)	Breaking Load kg (lbs)	Elongation %	Tensile Str. kg cm^{-2} (psi)
1A	None	3	2.54 (1.0)	.0254 (.010)	95 (200)	124	1400 (20,700)
2A	1×10^6	3	2.54 (1.0)	.0254 (.010)	84 (185)	150	1305 (18,650)
3A	1×10^7	3	2.54 (1.0)	.0254 (.010)	82 (180)	114	1270 (18,000)

The most apparent change in these samples of polyester films is a notable correlation between increasing radiation and decreasing breaking load and tensile strength. There is a net loss in elongation.

Table III. Radiation Effects on Polyester Film.

Spl. No.	Total Dose(R)	No. of Specs.	Width cm(in.)	Thickness cm(in.)	Breaking Load kg(lbs)	Elongation %	Tensile Str. kg cm^{-2}(psi)
1C	None	3	2.54 (1.0)	.0076 (.003)	28 (63)	58	1509 (21,160)
2C	1×10^6	3	2.54 (1.0)	.0076 (.003)	28 (62)	63	1460 (20,900)
3C	1×10^8	3	2.54 (1.0)	.0076 (.003)	27 (60)	81	1395 (19,930)

The samples in Table III showed that, as radiation increased, the tensile strength decreased and the elongation percentage increased. Possibly these property changes are caused by predominance of chain scission over crosslinking.

Table IV. Radiation Effects on Polyester Film.

Spl. No.	Total Dose(R)	No. of Specs.	Width cm(in.)	Thickness cm(in.)	Breaking Load kg(lbs)	Elongation %	Tensile Str. kg cm^{-2}(psi)
1D	None	3	2.54 (1.0)	.00254 (.001)	8.6 (19)	97	1320 (19,000)
2D	1×10^6	3	2.54 (1.0)	.00254 (.001)	7.1 (16)	31	1110 (15,800)
3D	1×10^8	3	2.54 (1.0)	.00254 (.001)	7.3 (16)	20	1140 (16,200)

Table IV shows that at 1×10^6R there is a lowering of tensile strength and breaking load coupled with an increase in elongation. At 10^8R, however, the breaking load and tensile strength are both higher, while the elongation has decreased by 10 per cent. These latter changes strongly suggest a predominance of crosslinking over chain scission (Fig. 1b).

The three polyester films shown in Tables II-IV are identical in all respects except thickness. Apparently the difference in the radiation effects which these materials exhibit is related to the thickness of the film. It has been suggested in the literature (Bolt and Carroll, 1963) that the thinner polyester films are the least vulnerable to damage by radiation. This specific contention was neither proven nor disproven by this study, but, as was pointed out in the previous discussion, there were variations in property changes which occurred in these materials related to sample thickness.

Table V. Radiation Effects on Flat-Conductor Cables
(Cable type 50614A. Tape)

Spl. No.	Total Dose(R)	No. of Specs.	Width cm(in.)	Thickness cm(in.)	Breaking Load kg(lbs)	Elongation %	Tensile Str. kg cm^{-2}(psi)
1A	None	4	1.36 (.5380)	.0254 (.01)	42.0 (92)	99	1200 (17,170)
2A	1×10^6	4	1.36 (.5330)	.0254 (.01)	40 (89)	62	1170 (16,750)
3A	1×10^8	2	1.34 (.5250)	.0254 (.01)	37 (81)	66	1030 (14,790)

2. Flat-conductor cable (Mylar). The changes shown in Table V include steady declines in breaking load and tensile strength. Elongation declines to a minimum at 10^6R and increases slightly with increasing radiation. These data appear to be somewhat similar to the effect illustrated in Figure 1b in which crosslinking is the predominant effect.

Table VI. Radiation Effects on Flat-Conductor Cables
(Methode Plyoduct, PD825P4)

Spl. No.	Total Dose(R)	No. of Specs.	Width cm(in.)	Thickness cm(in.)	Breaking Load kg(lbs)	Elongation %	Tensile Str. kg cm^{-2}(psi)
1A	None	3	1.4 (.5310)	.03 (.0125)	54 (110)	-31	1255 (17,940)
2A	1×10^7	4	1.4 (.5330)	.03 (.0115)	49 (108)	71	1240 (17,720)
3A	1×10^8	4	1.4 (.5250)	.03 (.0120)	47 (103)	59	1190 (17,000)

Just as with the other type of flat-conductor cable, the material illustrated on Table VI experienced a general decrease of tensile strength and breaking load with increasing radiation. A sharp increase in elongation percentage is noted up to 10^7R following which elongation percentage declined. These data suggest that chain scission was the dominant influence until at least 10^7R had been absorbed.

3. Teflon.

Table VII. Radiation Effects on Teflon

Spl. No.	Total Dose(R)	No. of Specs.	Shore Hardness	Breaking Load	Elongation %	Tensile Strength kg cm^{-2} (psi)
1A	None	4	48	7	434	185 (2640)
2A	1×10^5	-	57	Sample destroyed	Sample destroyed	Sample destroyed
3A	1×10^6	-	54	"	"	"
4A	1×10^7	-	Sample destroyed	"	"	"

The data in Table VII show the vulnerability of Teflon to radiation in an oxygen atmosphere. Almost immediately after irradiation began, the sample became too brittle to measure for all parameters except Shore hardness. Even this parameter lost its meaning at 1×10^7R because of the extreme embrittlement of the material.

Teflon is among the organic materials most sensitive to radiation in the presence of oxygen. Tensile strength is reduced to half its initial value at 4×10^6R, and elongation is lost at 2×10^6R. Degradation results from scission and is reported to be much less in vacuum than in air. Future studies should be directed toward testing in vacuum using samples of different thickness. It can be anticipated that thicker samples in vacuum will display significantly higher tolerance to radiation.

B. POTTING COMPOUNDS

Three types of potting compounds were tested in the current series of tests. These include polyurethane, silicone, and epoxy resins. Figures 4 through 8 show the effect of radiation and the compressive strength of these materials.

1. Epoxy resins. The type of curing agent used in curing an epoxy resin has a large effect on the radiation stability of the final product. In general, epoxy resins of high heat-distortion temperatures are more resistant than those having low heat-distortion temperature. In addition, aromatic type curing agents offer the most resistance to radiation.

The particular epoxies irradiated during this study(illustrated in Figures 4 and 5) showed the following significant changes: (1) With no. 1 Stycast 2850* GT Epoxy, the unirradiated sample received approximately 2150 kg (4750 lbs) compression in five seconds before failure. Samples of this material irradiated with 10^5R gamma radiation failed in 5 seconds with only 1900 kg (4200 lbs) compression while a third sample irradiated 1.5 x 10^6R failed at 1800 kg (4000 lbs) compression. Comparisons in Rockwell hardness illustrated in Table VIII showed a steady decrease in hardness from 77 to 49 as the radiation dose increased. These data suggest that degradation is caused by steady chain scission resulting in a decrease in compression strength and hardness. (2) Samples no. 6 of 15 per cent Flex C-1 Epoxy* showed an initial increase in compression strength at 10^5R gamma radiation followed by a decrease in strength and premature failure at 1.5 x 10^6R. Hardness data on these samples showed a slight increase in hardness at 10^5R and 100 per cent increase in hardness at 1.5 x 10^6R. Hardness data alone suggest that crosslinking is a dominant influence. This theory is corroborated by the initial compression strength test illustrated on curve 6B in Figure 1. However, curve 6C does not conform to this general pattern, which indicates that further study is required on this material. (3) Samples no. 5 of 75 per cent Flex C-1 Epoxy** showed a slight decrease in compression strength at 10^5R gamma radiation. Shore hardness data on this material showed an initial decrease at 10^5R followed by a sharp increase at 1.5 x 10^6R. These data suggest that there was initial degradation of this material caused by chain scission followed by crosslinking which caused an increase in hardness. (4) Sample no. 2 Stycast 2651 Epoxy* illustrated in Figure 5 showed a gradual decrease in compression strength at a dose of 10^5R followed by a sharp decrease at 1.5 x 10^6R. Hardness data showed a slight decrease in hardness at 10^5R followed by recovery to the original hardness at 1.5 x 10^6R. At best this data are inconclusive; but based on compression strength data, it can be postulated that chain scission is the dominant process. (5) Samples no. 3 of XR 5038 Epoxy* showed an increase in compression strength as radiation increased from 10^5R to 1.5 x 10^6R. At the same doses, the hardness value increased only slightly. This data suggest an increase in crosslinking because of radiation.

2. Silicone resins. Radiation affects this material by causing an increase in hardness and decrease in tensile strength and flexibility at 1 x 10^6R and higher. Dielectric properties are somewhat better and can stand doses as high as 9 x 10^8R before receiving appreciable damage.

The silicones studied showed the following significant changes: (1) Sample no. 7 EC 1663 in Figure 6 showed a gradual increase in compression strength as the amount of radiation absorbed by the material increased. Shore hardness showed no significant changes. The increase in compression strength

* All samples were 1/2" diameter x 1" length.

** All samples were 1" diameter x 1" length.

is small, but probably significant; it suggests that a slight crosslinking has occurred within the material. (2) Samples no. 9 and no. 11 LTV 182** and 602** showed no significant trends because of gamma radiation. Hardness values on these materials were too close to be significant. (3) Samples no. 12 RTV 11 Silicone** showed an initial increase in compression strength at 10^5R followed by a decrease in strength at 1.5×10^6R (Fig. 7). Hardness values on this material showed a slight decrease as radiation increased. This data are somewhat inconclusive, but it may suggest that a trend toward chain scission is the predominant effect. (4) Samples no. 10 RTV 881 Silicone** showed only slight variations from the unirradiated material. These changes indicate a slight decrease in strength at 10^5 followed by an increase at 1.5×10^6R. Hardness values indicate an initial slight decrease at 10^5 followed by a return to the original value at 1.5×10^6R. The decrease in strength and hardness at 10^5 may be caused by initial scission reactions followed by a predominance of crosslinking at 1.5×10^6R.

3. Polyurethane resins. These resins exhibit a high degree of resistance to radiation and are reported to be surpassed only by aromatic-cured epoxy resins and phenolic resins in radiation resistance. The radiation resistance of this material is adversely affected by the presence of water which increases the probability of damage.

The polyurethanes illustrated in Figure 8 showed the following trends: (1) Samples no. 4 of Eccofoam FPH Polyurethane** showed a marked decrease in compression strength as radiation increased. This data may indicate a trend toward chain scission of the molecules. (2) Samples no. 8 PR 1538** showed a steady decline in compression strength coupled with a decrease in hardness as radiation increased. This data also suggest that a dominance of chain scission was occurring in the molecules.

Table VIII. Radiation Effects on Hardness Properties of Selected Potting Compounds

	Shore Hardness		
	OR	1×10^5R	1.5×10^6R
Epoxy C1 75% Flex	80	60	>100
Polyurethane 1538	76	75	70
Silicone RTV11	47	45	42
Silicone LTV182	27	29	30
Silicone LTV602	21	17	18
Silicone EC1663	47	43	47
Silicone RTV881	38	35	37
	Rockwell Hardness (E Scale, 100 kg load, 1/8" ball)		
Epoxy 2850GT	77	66	49
Epoxy 2651	56	49	57

** All samples were 1" diameter x 1" length

Table VIII. Effect of Radiation on Hardness Properties of Selected Potting Compounds (Cont'd)

	Rockwell Hardness (H Scale, 60 kg load, 1/8" ball)		
	OR	1×10^5R	1.5×10^6R
Epoxy XR5038	62	60	63
Epoxy C1 15% Flex	15	18	39

CONCLUSIONS

Some of the problems inherent in relating radiation effects data to changes in physical properties of polymeric materials have been discussed. Analogies were drawn between data obtained in a recent series of tests conducted at Oak Ridge, Tennessee, and published information on radiation effects on organic materials. Interpretations of the meaning of changes in compression strength, elongation, etc., in terms of changes in molecular weight, chain scission, and crosslinking were made only after due regard to theoretical considerations.

The data presented are considered tentative because sufficient samples were not available to obtain statistical averages. However, it is anticipated that the new data will change only slightly when statistical averages are used. In addition, the tests reported are preliminary to a more complete series of space environmental tests to be conducted in the near future.

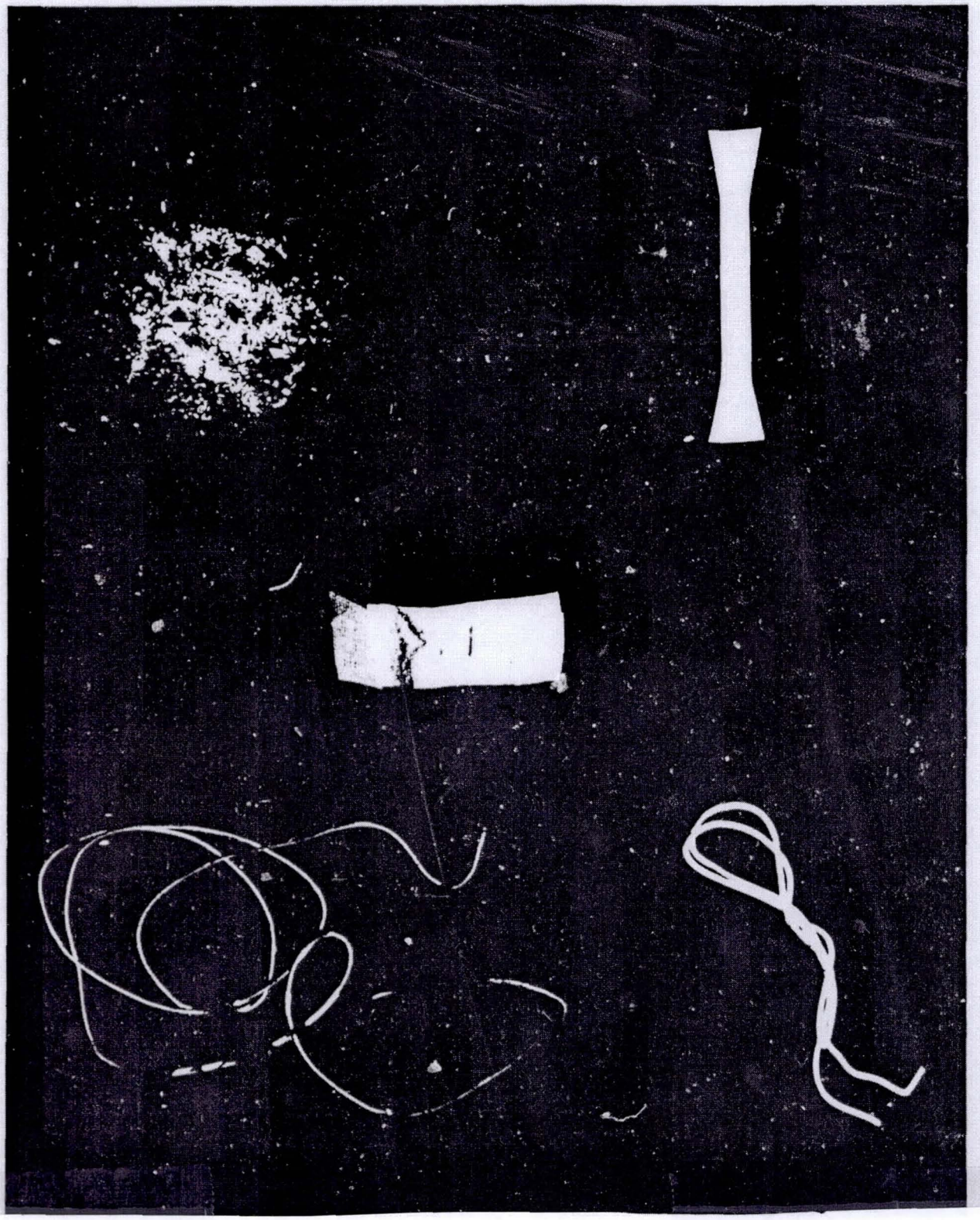


FIGURE 2. RADIATION EFFECTS ON TEFLON AND COPPER FOIL. UPPER LEFT, TEFLON FILM, 10^6R; UPPER RIGHT, TEFLON FILM, 0 R; CENTER, COPPER FOIL, 10^8R (note loss of adhesive); LOWER LEFT, TEFLON WIRE INSULATION, 10^8R; LOWER RIGHT, TEFLON WIRE INSULATION, 0 R.

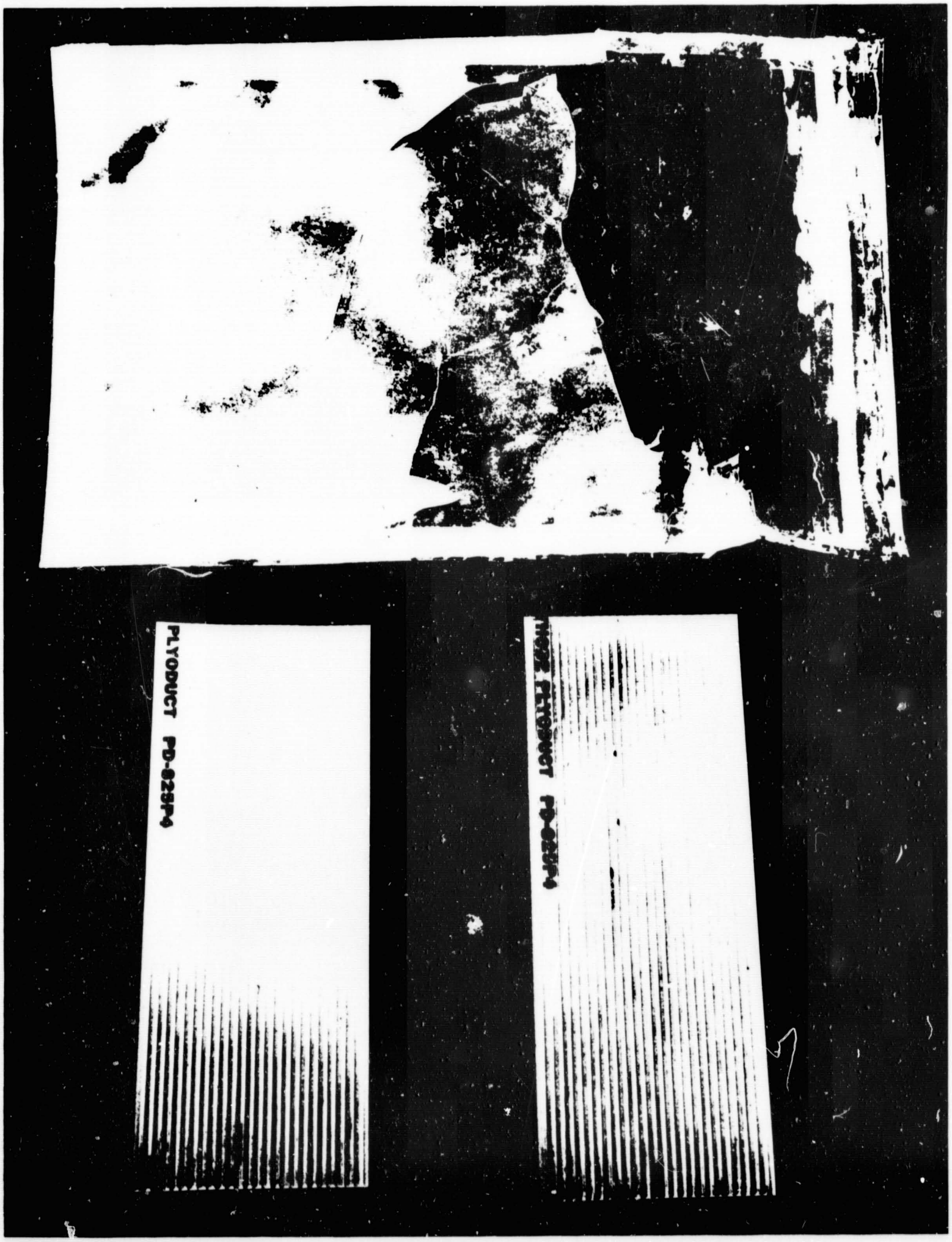


FIGURE 3. RADIATION EFFECTS ON FLAT-CONDUCTOR CABLE AND POLYETHYLENE BAG. UPPER HALF, POLYETHYLENE BAG, 10^8R; LOWER LEFT, FLAT-CONDUCTOR CABLE, 10^8R; LOWER RIGHT, FLAT-CONDUCTOR CABLE, 0 R.

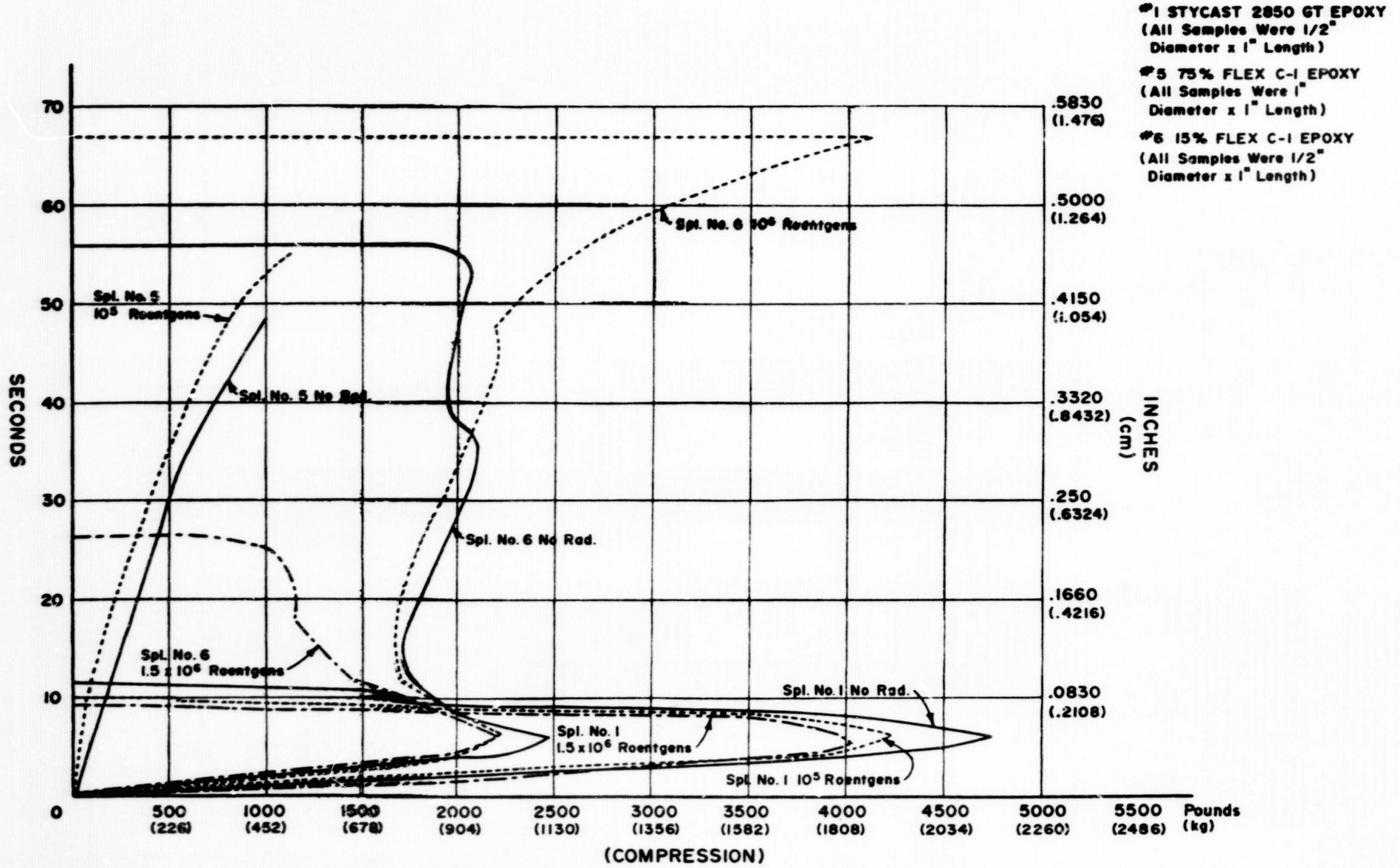


FIGURE 4. RADIATION EFFECTS ON EPOXY RESINS

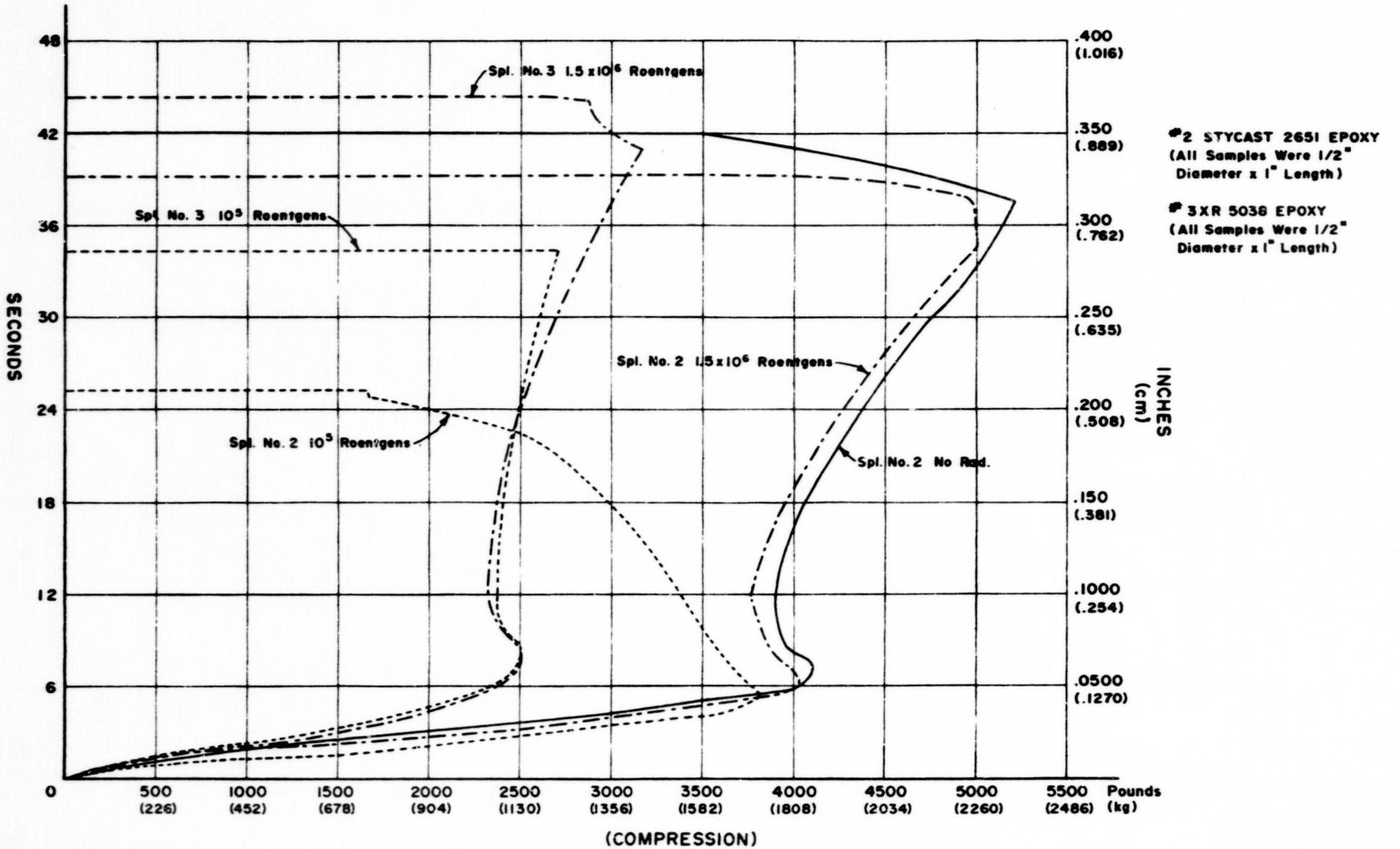


FIGURE 5. RADIATION EFFECTS ON EPOXY RESINS

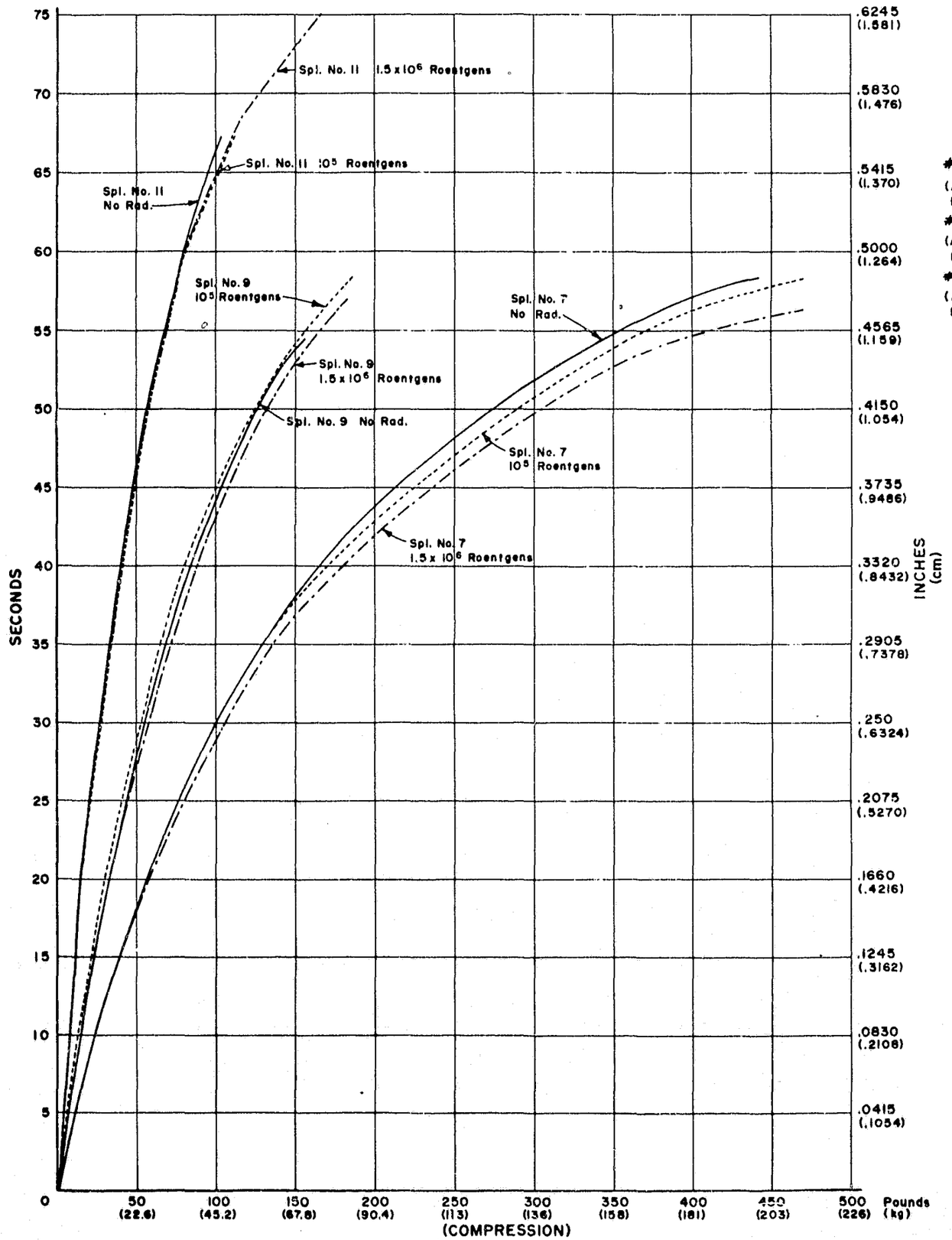


FIGURE 6. RADIATION EFFECTS ON SILICONE RESINS

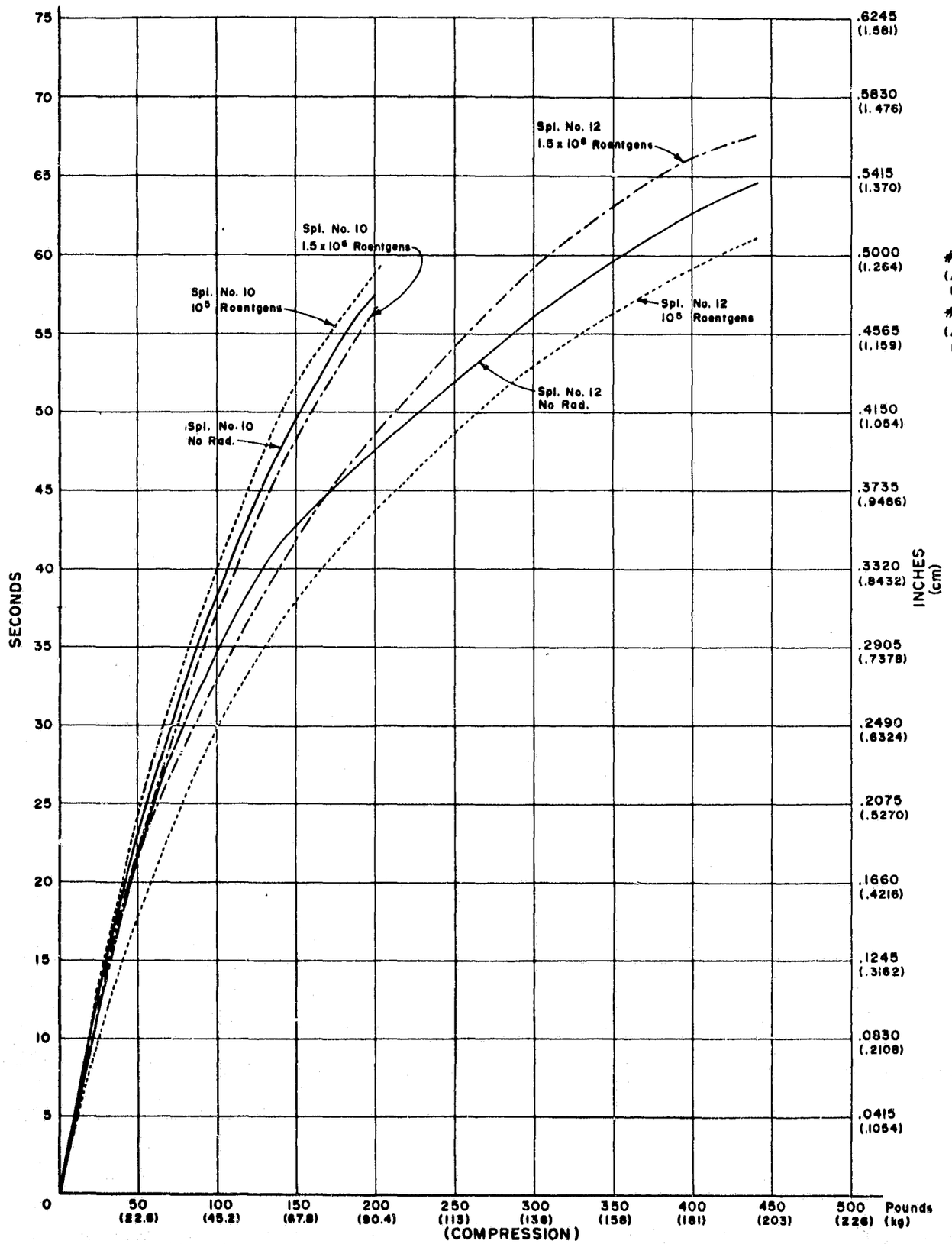


FIGURE 7. RADIATION EFFECTS ON SILICONE RESINS

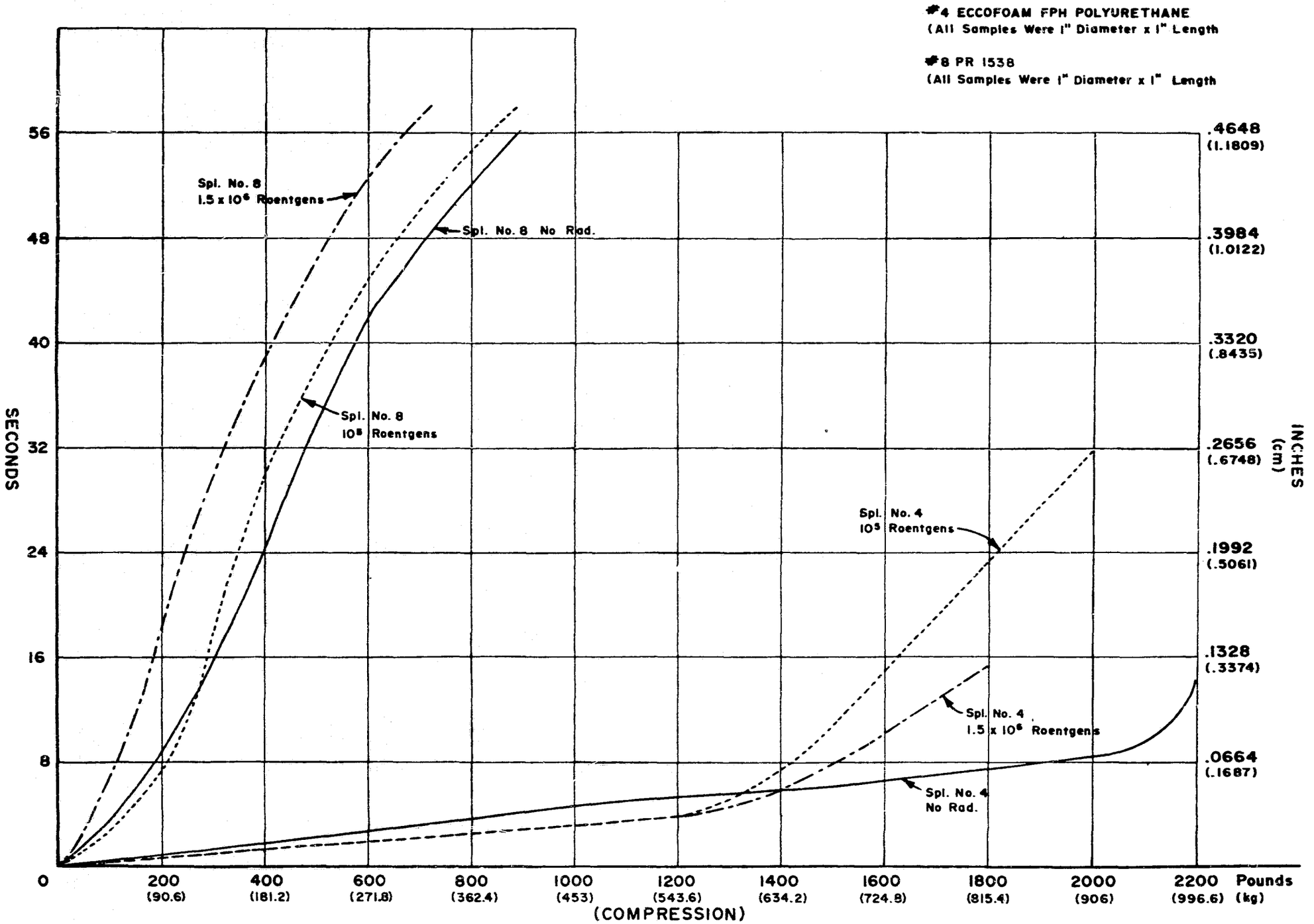


FIGURE 8. RADIATION EFFECTS ON POLYURETHANE RESINS

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APPROVAL

MTP-ASTR-P-63-16

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by

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The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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